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LITERATURE SURVEY OF INSTRUMENTAL MEASUREMENTS OF BIOCHEMICAL OXYGEN DEMAND FOR CONTROL APPLICATION 1960-1973

NATIONAL ENVIRONMENTAL RESEARCH CENTER

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16. ABSTRACT

Development of a rapid, instrumental method for determining waste loading in terms of biochemical oxygen demand (BOD) would further the efforts of controlling the unit processes of sewage treatment. This report attempted to determine the "state of the art" of instrumental biochemical oxygen demand methods through a survey of related literature that included material published between 1960 and 1973. The slow rate of microbial reactions made the task of an instrumental approach to BOD difficult. Microorganism variability in numbers, kind, and acclimation caused a lack of reproducibility. Although the present "state of the art" does not permit instrumental measurement of BOD for process control, an alternative solution is suggested for secondary treatment plants. Further research is needed to supplant the BOD test with this method. A process needs to be determined (e.g., biofiltration and recirculation) to reduce the BOD of a secondary effluent sample to a sufficiently low value. Differential measurements (ΔTOC, ΔTOD, ΔCOD) of the secondary effluent and the processed sample produces a good estimate of the ultimate BOD. Successful efforts in this research would produce greater operating efficiency and reduction in pollution discharge to receiving streams by the waste treatment plants.

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REVIEW NOTICE

The National Environmental Research Center -- Cincinnati has reviewed this report and approved its publication. Approval does not signify that the contents necessarily reflect the views and policies of the U. S. Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

FOREWORD

Man and his environment must be protected from the adverse effects of pesticides, radiation, noise and other forms of pollution, and the unwise management of solid waste. Efforts to protect the environment require a focus that recognizes the interplay between the components of our physical environment--air, water, and land. The National Environmental Research Centers provide this multi-disciplinary focus through programs engaged in

- studies on the effects of environmental contaminants on man and the biosphere, and
- a search for ways to prevent contamination and to recycle valuable resources.

This report is part of a continued effort by the Instrumentation Development Branch, Methods Development and Quality Assurance Research Laboratory, NERC, Cincinnati, to evaluate instruments and provide information to both users and suppliers. It is also intended that instrumentation be upgraded and that a choice of the most suitable instrument can be made for a particular application.

A. W. Breidenbach, Ph.D.
Director
National Environmental
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SECTION I

CONCLUSIONS

A most urgent need in the waste treatment field is that of an on-line biochemical oxygen demand (BOD) instrument suitable to control the treatment process. The slow rate of microbial reactions makes the task of an instrumental approach to BOD difficult. The BOD test is losing its status as a standard method; lack of reproducibility because of microorganism variability in numbers, kind, and acclimation are the reasons that BOD test results are questioned. Therefore, a new standard, amenable to instrumental measurement, is critically needed to supplant the BOD test for the measurement of waste water loading. In addition, the standard should be independent of microbiological activities and their inherent uncertainties, although it must show the results of biological action because, presently, secondary treatment plants are almost exclusively biological.

Differential measurements, such as differential chemical oxygen demand (ΔCOD), in conjunction with further treatment of sampled effluents of treatment plants, will show the effects of biological action. The differential indications are a result of organic wastes being removed by the degradation and assimilation of microorganisms in the treatment process. More research effort is needed on the differential measurement approach so that the BOD test can be supplanted as the standard of waste load, and the information of the BOD test can be obtained more directly, rapidly, and reproducibly.

SECTION II

RECOMMENDATIONS

The approach contemplated for this literature survey was to relate ΔCOD measurements across a treatment plant to methods of process control. Although papers by Gaudy[19,20] and others indicated that this approach was not unique, the practice does not appear to be widespread, possibly because further refinement is necessary. The ΔCOD does give a measurement related to the organic matter removed by microorganisms in the treatment plant, but it does not give any estimate of the total organic matter in the waste to begin with. The efficiency of waste removal is a variable to be determined .tself; therefore, more information is needed. More research in this area could lead to ways to further remove BOD from a sampled effluent. If the rate and extent of BOD removal is acceptable, a very good estimate of the ultimate BOD can be made by adding the ΔCOD measured across the process to that measured across the treatment plant. This differential approach may also be applied to total organic carbon (TOC) and total oxygen demand (TOD). In fact, the TOC would be the measurement of choice because all of the carbon of a sample is converted to carbon dioxide without interferences. Incomplete combustion occurs for some nitrogen and sulfur compounds in the instrumental measurement of TOD and COD. The effect, however, of the incomplete combustion is reduced because the measurement of difference is being made. Therefore, it is recommended that various biofiltration media be tested and that ΔTOC, ΔTOD, ΔCOD, or all three measurements be made and analyzed for correlation to the BOD dilution test. The intent is to provide a sample process amenable to inclusion in a control approach for waste treatment plants.

Automation of these differential measurements would involve various sampling devices and their control, analog to digital converters, storage registers, and other digital logic techniques. Servo-mechanism techniques, or direct digital control would be adaptable for controlling elements of the individual processes. Process control ability can be demonstrated, initially, in a pilot treatment plant.

SECTION III

INTRODUCTION

The intent of this literature survey, which includes material published between 1960 and 1973, is to determine the "state of the art" of instrumental biochemical oxygen demand (BOD) methods. The ultimate goal is an instrument capable of controlling the waste treatment process. Such an instrument must detect waste loads and indicate the effectiveness of the waste treatment process in removing the waste.

Sources of information for this survey have included textbooks concerning waste treatment, U.S. Environmental Protection Agency (EPA) publications and training manuals, journals in the environmental and waste treatment field, and bibliographic references. Of particular usefulness was a related abstract search.[28] The Water Resources Abstract publication of the U.S. Department of the Interior was also very useful. Background information was obtained on the microbial, mathematical, and chemical aspects of BOD as well as on instrumental approaches to its measurement. Some pertinent background information is not specifically referred to elsewhere in the text.[13,21,31,34,37,40,44] The NERC - Cincinnati Library was most helpful in obtaining copies of the related papers.

SECTION IV

BACKGROUND

The development of an effective and practical control scheme for treating municipal sewage is hindered by the inability to measure BOD on-line.[2,4,24,25] Although the BOD test has many disadvantages, it is the conventional standard of waste load measurement.[12,14,36] Development of a rapid, instrumental method for determining waste loading in terms of BOD or its equivalent would further the efforts of controlling the unit processes of sewage treatment. This control would result in greater plant operating efficiency. The resultant reduction in pollution discharge to receiving streams would enhance their quality significantly. A direct, rapid, reproducible measurement of waste loading, then, is required for the often-stated goal of EPA and the waste treatment field -obtaining an on-line BOD sensor.

SECTION V

BOD TEST

BOD is usually defined as the amount of oxygen that bacteria require while stabilizing decomposable organic matter under aerobic conditions.[36] Dissolved oxygen (DO) is the energy source of these microorganisms when they act upon the organic waste. In Standard Methods for the Examination of Water and Wastewater,[1] it states that BOD determination is an empirical test used to estimate the relative oxygen requirements of wastewaters, effluents, and polluted waters and that its most frequent use is in measuring waste loadings of treatment plants and in evaluating the efficiency (BOD removal) of such treatment systems. Since complete stabilization of a given waste is not feasible, a 5-day standard is used to obtain a practical estimate of the BOD. Mathematical relationships based upon a first-order[38] chemical reaction are used in determining the ultimate BOD. Dilution commonly provides the excess oxygen required for heavy oxygen demanding wastes. An acclimated seed from a receiving stream is generally employed to furnish microorganisms for the test.

The BOD test is losing its status as a standard because of the difficulty encountered in controlling the variables involved: time, seeding, dilution, temperature, pH, and toxic substances. In addition, the first-order assumption is presently recognized as an oversimplification.[1,18] The variability and acclimation of microorganisms used in seeding are given as the primary cause for lack of accuracy and reproducibility of interlaboratory BOD tests on known, single source samples. Process control using the BOD measurement is completely ruled out because of the extensive time

required to complete the test. Many sources, however, have indicated the urgent need for an on-line instrumental measurement of BOD.[2,4,24,25] Perhaps, this controversy over the BOD is put in its proper perspective by A. F. Gaudy, whose recommendation will be discussed more fully later.[19,20]

> "It is necessary to make a distinction between the concept and the test (BOD test). While we can defend and recommend the concept, we shall not defend the test for all its present applications, but will recommend a more satisfactory measurement for assessing efficiency of treatment by the activated sludge process."

SECTION VI

INSTRUMENTAL BOD

An automated version of the BOD test can be obtained by employing respirometers. Numerous versions are described in the literature by authors in the United States and abroad.[3,5,17,22,39,42,43] The fundamental concept is adding either air or pure oxygen to microorganisms in a reaction chamber to replenish the oxygen consumed. One version generates oxygen, upon the sensed requirement, by means of electrolysis.[9,47,48,49,50] The oxygen consumption is generally measured manometrically through the difference in pressure or by means of a DO sensor. A caustic solution, usually potassium hydroxide, is placed in the air circulation stream to absorb carbon dioxide. Various transducer devices are usually employed to record the potentiometric output on a strip chart.

Originally, respirometers were manually operated by highly trained technicians capable of interpreting the derived results analytically. The operation has been simplified over the years, and the outputs have been related to BOD directly.

Respirometric methods are an improvement upon the dilution test in the sense that generally the oxygen uptake is obtained directly from the undiluted sample; however, time, seeding, temperature, pH, and toxic substances remain as variables that will affect the results as they do in the standard BOD dilution test. Recently, the time factor has been assailed by relating the short-term oxygen uptake to the BOD test.[30] An improved approach is to relate the impact of a waste sample upon the endogenous respiration rate of a suitable activated sludge:[45] the slope change

encountered in oxygen consumption is correlated to BOD loads. However, short-term BOD tests, utilizing respirometers, are based upon a relationship, to the dilution test; i.e., they are being based upon a test that is already of questionable value. As in the dilution test, microorganism variability render the results suspect. The respirometer utilizing these short-term techniques does, however, serve a need for determining, rapidly, the treatability or toxic nature of a waste.

SECTION VII

RELATED ORGANIC POLLUTION MEASUREMENTS

Other measurements of pollution load are chemical oxygen demand (COD), total organic carbon (TOC), and total oxygen demand (TOD). The COD test, although determined more rapidly than BOD, still requires 2 or more hours to obtain meaningful results. TOC and TOD are instrumental measurements requiring 5 minutes or less for results. Automated wet chemical procedures have been perfected for the measurement of COD.[26,29,46] The primary disadvantage of all these measurements is that the biological nature of the waste treatment plant loads are not characterized.

The COD test is a measure of those organic materials oxidizable by dichromate (a strong chemical oxidant) in acid solutions at high temperature.[1] Some organic compounds (e.g., acetic acid) require a catalyst to be oxidized. Ammonia is not oxidized by dichromate at all. This is important because acetic acid and ammonia are oxidized biologically. Other organic compounds (e.g., cellulose) are oxidized in the COD test although they do not present an immediate biochemical load to a receiving water. An undersirable aspect of the test is the requirement to handle hazardous chemicals. When applicable, however, the COD test is preferred to the BOD because it is more rapid and reproducible. Most organics are oxidized to 95 to 100 percent of theoretical amount in the COD test.[10] A comparison of recent rapid methods and past methods of determining COD with the method of present choice in Standard Methods shows the latter to be, by far, the most accurate.[16]

The TOC measurement is obtained instrumentally by an operator

first measuring the total carbon and then substracting a separate measurement of the inorganic carbon.[15,32] Oxygen gas is passed continually through a combustion chamber containing a cobalt oxide catalyst. A micro sample is injected into the chamber, which is maintained at 950°C. The sample is vaporized, and all the carbonaceous material is oxidized. The oxygen carrier gas transports the carbon dioxide and steam from the chamber. An infrared analyzer measures the carbon dioxide present; the amount present, which is in the form of a peak on a strip-chart recorder, is proportional to the total carbon of the injected sample. Output calibrations are performed with samples of known carbon content. A low temperature combustion chamber is operated in parallel at 150°C, a temperature below that that oxidizes organic matter. An identical micro sample is injected in this chamber over quartz chips coated with phosphoric acid. Inorganic carbon is oxidized to carbon dioxide. An oxygen carrier gas, again, conducts the carbon dioxide to the infrared analyzer for measurement. This value substracted from the total carbon previously determined results in a value for the TOC.

TOC is considered one of the fundamental measurements of pollution.[15] When performed properly, TOC determinations can be made reliably, rapidly, and reproducibly. Some disadvantages are the initial instrument cost and the requirements of operator experience and technique to obtain reproducible results.

Another instrumental measurement is TOD,[10,23] the quantitative measure of oxygen required to combust organic matter of a sample at high temperature. A nitrogen carrier gas with a fixed amount of oxygen (usually 200 ppm) flows continuously through a combustion

chamber that is maintained at 900°C and contains a platinum catalyst. The oxygen in the carrier stream is continuously measured in a silver-lead fuel cell detector. A continuous output on a strip chart recorder results from this equilibrium condition. An aqueous sample with impurities injected into the combustion chamber will disturb this equilibrium. The impurities are oxidized by partially depleting the oxygen on the platinum surface. The oxygen in the carrier gas replenishes the oxygen on the platinum surface. This momentary demand on the oxygen in the carrier gas is detected by the silver-lead fuel cell, and a negative going peak (or trough) is recorded. The measurement consists of comparing the peak heights with calibration of known sample contents.

Complete combustion of carbon and hydrogen takes place during the measurement of TOD. Nitrogen compounds are oxidation-state dependent where molecular nitrogen (the carrier gas) is unaffected. Sulfur compounds are only partially oxidized. DO contributes to the oxidation process and thus reduces the measured demand equivalent to the milligrans of DO per liter of sample. A very close correlation between TOD and COD has been demonstrated on several types of effluent.

SECTION VIII

CORRELATIONS BETWEEN BOD, COD, TOC, AND TOD

Utilizing correlations between BOD and COD has been discussed in the literature.[35,46] Some sources[11,15] qualify their use only to fixed effluents such as, the industrial effluent from an unchanging process. The consensus is that for treatment of mixed industrial and municipal wastes the BOD load varies over a wide range of values. The COD also ranges over a considerable span, but these variations in BOD and COD are not in the same ratio with each other. Municipal waste treatment loads vary from week to week, day to day, and even from hour to hour. Individual measurements of BOD, COD, TOC, and TOD can tell a lot about the characteristics of a waste, but no attempt should be made to replace BOD measurements with COD, TOC, or TOD. Ford[15] listed factors to consider when attempting to use correlations between the various measurements.

> "In attempting to correlate BOD or COD of an industrial waste with total organic carbon concentration one should recognize those factors which may discredit the correlation. These limitations include: 1) a portion of the COD of many industrial wastes is attributed to the dichromate oxidation of ferrous iron, nitrogen, sulfites, sulfides, and other oxygen consuming inorganics; 2) the BOD and COD tests do not include many organic compounds which are partially or totally resistant to biochemical or dichromate oxidation (however, all of the organic carbon in these compounds is recovered in the TOC

analysis); and 3) the BOD test is susceptible to variables which include seed acclimation, dilution, temperature, pH, and toxic substances."

SECTION IX

DIFFERENTIAL MEASUREMENTS

Gaudy[19,20] has strongly recommended replacing the BOD test with the ΔCOD measurements across an activated sludge process. Others have discussed similar use of the COD in the literature,[6,27,33,41] but widespread acceptance has not been indicated. The ΔCOD technique may be practiced in the field without a great deal of publicity. Nonetheless, Gaudy defined ΔCOD as "the amount of COD removed at any time, i.e., the difference between the COD present at the time of measurement and the COD initially present." The organic matter removed from a waste treatment plant is, then, the difference between the effluent COD and the influent COD.

$$\Delta COD_r = COD_i - COD_e \qquad (1)$$

where

ΔCOD_r = metabolizable organic wastes removed by microorganisms used in the process

COD_i = plant influent COD

COD_e = plant effluent COD

Gaudy stated that the day-to-day operation of the waste treatment process can be assessed with a determined efficiency of COD removal:

$$\text{efficiency of COD removal} = \frac{\Delta COD_r}{COD_i}$$

Another operational parameter is that of the COD_e, which is continually compared with the base line COD_e established in treatability studies performed before the design stage. In

addition to the operational parameters, Gaudy recommended periodical sampling of the influent waste in a batch laboratory reactor. COD tests are performed on this sample until changes in COD no longer occur. The ΔCOD of this sample gives a direct assessment of the actual purification achieved when compared with that of the ΔCOD across the treatment plant.

Busch[8] indicated that the change in TOC measurements due to bacterial action (similar to ΔCOD) is a prime parameter in water pollution control work. Also, process control capability was indicated by "a mass culture aeration procedure for a rapid (1 to 4 hours) determination of biodegradable carbon content." Busch expressed this differential as total bacterially available carbon (T_bAC). Busch[7] advocated using ΔTOC measurement along with short-term (24 hour) BOD measurements to obtain the total biological oxygen (T_bOD). Busch contended that the increase in bacterial mass must also be accounted for in the use of oxygen consumption. This results in some simple-to-complex combination measurements of varying accuracies and time intervals to account for the materials balance concept.

SECTION X

DIFFERENTIAL MEASUREMENTS FOR PROCESS CONTROL

This literature survey has not revealed a direct, rapid, and reproducible instrument or method of determining BOD that is suitable for process control. The literature has instances of respirometers successfully monitoring sewage treatment influent/effluent wastes.[22] Short-term oxygen demands are related to 5-day BOD's. The suitability of indirect estimates of BOD by respirometers for process control is questionable. As in the dilution test, results depend on microbial reactions, which are not reproducible in a measurement sense.

Since the status of the BOD test as a standard is waning, an alternative test should be pursued. The BOD test has had its widest application in determining waste loadings and efficiencies in waste treatment plants. By 1977, municipal waste treatment plants are required to use secondary treatment, and differential measurements such as ΔCOD, ΔTOC (i.e., T_bAC), and ΔTOD all can be applied across these secondary waste treatment plants to obtain useful information. Refinements and further research, however, are needed before differential measurements can be used for process control.

Before differential measurements can be applied to process control, a better estimate of the total biologically degradable organic waste must be made, i.e., each measurement must include an estimate of this total metabolizable material so that process modifications can be made intelligently. If the waste removed and a good estimate can be made of the maximum waste that can be

removed are both known, then the process efficiency can also be determined. It is herein theorized that a sufficiently accurate estimate of the total metabolizable waste is obtained if a small sample is processed after secondary treatment so that complete removal of BOD is approached. A differential measurement across this processed sample, when added to the differential across the secondary treatment plant (i.e., from influent to primary) will give this estimate. Optimally, simple biological filtration of a sample, settling, recirculation, and subsequent passage through a 0.45μ filter will approach the complete removal of BOD. The development of this process, utilizing various filter media, would be the subject of further research in this area. Monitoring the recirculated sample for several of the instrumentally measurable basic parameters (DO, pH, ORP, temperature, turbidity, and ammonia, if available) would also be of interest. One of these parameters could be an indicator of the completion of the process. If the biofiltration approach is unsuccessful, biological suspension by aeration might be pursued. Initially, however, biofiltration would appear to be the simpler approach. Again, the ultimate goal is instrumental control of the waste treatment process.

Processing a secondary effluent sample to achieve an acceptable degree of BOD removal would make possible determining the waste discharge load to the receiving stream in terms of the differential measurement. For example, in terms of COD:

$$COD_e - COD_p = \Delta COD_d \qquad (3)$$

where

COD_e = plant effluent COD

COD_p = sample processed to approach zero BOD

ΔCOD_d = metabolizable organic waste in the plant effluent

Then the total organic matter of an influent waste that is decomposable by microorganisms is

$$\text{Total decomposable waste} = COD_r + COD_d \qquad (4)$$

The efficiency of the plant's waste removal operation is

$$\text{Plant efficiency} = \frac{COD_i - COD_e}{COD_i - COD_p} \times 100 \qquad (5)$$

$$= \frac{\Delta COD_r}{\Delta COD_r + \Delta COD_d} \times 100$$

Data obtained from the standard BOD and COD tests can be collected at various secondary treatment facilities to verify these relationships. In summary, equations (1), (3), (4), and (5) will enable the daily determination of:

1. waste treatment plant load in terms of ΔCOD_r
2. waste load discharged to the stream
3. the total oxygen demanding load of the influent to the plant
4. the efficiency of the treatment plant to remove metabolizable organic waste.

The use of COD values for equations (3), (4), and (5) is for illustrative purposes only; TOC, TOD, or instrumental COD can be used equally well, and their use may very well be better. In fact, if funding permitted, it would be well to purchase these instruments and compare all three methods. The chemical ΔCOD method is directly applicable to small waste treatment plants that cannot afford expensive instrumentation. The ultimate goal, however, is to automate the measurement for control purposes, and instrumental TOC, TOD, or COD may be more amenable to this. The sample-handling of TOD is already capable of continuous operation.[19]

SECTION XI

SUMMARY

Prospects for process control of waste treatment plants appear favorable if differential measurement capabilities can be developed further. If rapid, reproducible removal of BOD to an acceptable value can be accomplished, differential measurements (ΔTOC, ΔTOD, or ΔCOD) can give the information of the ultimate BOD in less than 1 day. Instrumental TOC, TOD, and COD can already be determined in less than 5 minutes. Detention times for secondary waste treatment would add 6 to 8 hours. The additional processing method and detention time would be the subjects of further research. The method of choice would be biological filtration in which various filtration media, and biological activation techniques would be pursued.

SECTION XII

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